Original Article

The optimization and characterization of the recycling utilization of raffinate in the copper leaching process

Long Wang, Shimin Song, Hanyu Gao, Ling Wang, Siyuan Yang, Cheng Liu

School of Mining Engineering, North China University of Science and Technology, Tangshan 063210, China
State Key Laboratory of Mineral Processing, Beijing 102600, China
School of Resources and Environmental Engineering, Wuhan University of Technology, Wuhan, Hubei 430070, China

ABSTRACT

The raffinate in the extraction process contains plenty of acid and metallic ions, the dispose of which causes the resource loss and environmental pollution. The present study proposes an innovative way to solve this problem by using raffinate as the leaching agent in the copper leaching process. The effects of significant leaching parameters were investigated and optimized using the central composite design (CCD) of response surface methodology (RSM). A maximum leaching efficiency of 96.74% can be obtained under the 53.22 Kg/t of initial acidity, 2.54 h of leaching time and 5.95 of liquid/solid ratio. In addition, the comparative features of samples before and after leaching process were analyzed by using the X-ray diffraction (XRD), sieving test, scanning electron microscope (SEM) and adsorption-desorption isotherms. We found that the majority of copper components have been removed from copper ores, causing the reduction of size distributions. Specifically, the content of <38 μm after leaching was largely increased from 43.71% to 85.00%. It also led to the appearance of micro pores and cracks in the copper ores, so as to increase the specific surface area and the adsorption mean aperture, which were beneficial to the copper leaching process.

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1. Introduction

Copper, one of the essential metals in the world, is extensively used in many areas for its excellent properties, such as electricity conductor, corrosion resistance and heat conductor [1-3]. The global copper mine production capacity reached 16 million tons in 2010, and grow quickly in the following years [4]. The majority of copper ores in the earth curst are in the form of copper sulfide or copper oxide which normally contain 0.5-2.0 % copper [5,6]. With the rapid depletion of relatively simple copper sulfide can be readily beneficiated, the low-grade copper oxide becomes the main raw materials for the copper products [7]. Recovering copper from the copper oxide ores using hydrometallurgical routes are widely used in prac-
tice for its advantages of high leaching efficiency, low costs and simple flow [8,9].

To obtain the high-quality cathode copper from leachate, the large amount of extracting agents are applied in the extraction process to purify the leachate, thereby increasing the concentration of copper ions in solution [10,11]. During the copper extraction, the raffinate is the liquid steam which remains after copper ions from the original leachate are removed through contact with the extracting agent [12]. The raffinate usually does not have enough commercial value and existed as the effluent due to its high acidity, thereby the plant need to maintain the wastewater treatment facility to deal with it [13].

Considering the raffinate still contains the relatively high amount of acid and metallic ions which were the by-products of extraction, the recovery and recycling of the raffinate have been attempted in recent years. Hydroxide precipitation is an effective method to recover metal ions from raffinate by adding lime or sodium hydroxide [14]. In addition, jarosite can be used to recover copper ion, zinc ion, cobalt ion and nickel ion from raffinate by forming jarosite complexes [15]. Ion exchange, which is of high selectivity, is another effective measure for recovering valuable ions from raffinate. Ion exchange and chemical precipitation were evaluated by some researchers in the removal of iron ions from copper solvent extraction and electrowinning process and ion exchange was identified to be a more efficient method [16].

Extensive researches have been conducted on the recovering of metal ions from raffinate by physical and chemical methods while most of them need to be neutralized with the acid in the raffinate or recover by some materials [17,18]. The cyclic utilization of raffinate in the leaching of low-grade copper oxide ore has been neglected for a long time. The present study attempts to apply the raffinate as the leaching agent and maximize the copper leaching efficiency using response surface methodology (RSM) which is an analytical technique applied in statistical modeling, experimental optimization and multivariable experimental design [19,20]. RSM is widely used for examining the relationship and confirming the influence between different experimental variables and factors [21–24].

The effects of leaching parameters and the optimum leaching conditions were investigated to reveal the possibility of using raffinate as the leaching agent in the copper leaching process. The features of copper ores before and after leaching process were analyzed by the X-ray diffraction (XRD), sieving test, scanning electron microscope (SEM) and adsorption-desorption isotherms.

2. Materials and methods

2.1. Materials

The low-grade copper oxide ore and raffinate were obtained from a hydrometallurgical plant located in Mulashes, Zambia. The size range of sample was 70 % below 74 µm. The chemical assay and components of the copper ores were analyzed by the X-ray fluorescence and X-ray diffraction, respectively, which revealed that the copper grade is low (1.53 %) and the main gangue mineral was the quartz. The main copper phase in the copper minerals was the copper oxide. Elements in raffinate were identified using atomic absorption spectroscopy (AAS), which revealed that the raffinate was the high concentration acid (15.3 g/L) coexist with some metal ions with low concentrations. More details of elements analysis of copper ores and raffinate were shown in Table 1.

2.2. Leaching tests

Leaching tests under the certain temperatures were carried out in a 1 L beaker with a controller of temperature and stirring speed. In each test, 100 g of low-grade copper oxide ore was leached with leaching time of 3 h, temperature of 65 °C and stirring speed of 500 rpm. To maintain the sufficient acid content, a required concentration of sulfuric acid (with respect to solid material) was added into the raffinate as the leaching agent. The heating was stopped, and the beaker was cool to the room temperature at the end of each test. Afterwards, the mixture was filtered, and the residue was washed with water and dried in oven. The content of copper in the residue was analyzed by AAS. Copper leaching efficiency (E) was calculated by

\[
E = \left[1 - \frac{(\text{Mr}/\text{Mo})}{100}\right], \quad (1)
\]

where Mr and Mo are the mass of copper in the residue and run-of-mine respectively.

2.3. Investigations and optimizations and of the leaching parameters

The module of central composite designs (CCD) in the response surface methodology (RSM) were adopted by using software “Design-Expert” to optimize the leaching process and to analysis the interaction between the parameters. A 2^3 CCD with 15 runs and 6 replications of the center points were chosen to obtain the optimum leaching conditions. According to previous experiments, initial acidity (X_1), leaching time (X_2) and liquid/solid ratio (X_3) chosen as the independent variables were given in Table 2, demonstrating the practical figures of the independent variables at which the experiment was carried out to evaluate the response variable (Y) [21,22].

The independent variables used in this experiment were coded as follows:

\[
X_i = \left(x_i - x_0\right)/\Delta x_i, \quad (2)
\]

where \(X_i\) is the coded values of the independent variable, \(x_0\) is the center point value of \(x_i\) and \(\Delta x_i\) is the step change value. According to CCD, the initial acidity varied from 18.4 to 55.2 Kg/t, the leaching time changed from 2 to 4 h, the liquid/solid ratio increased from 3 to 6. The optimization results of the leaching process were illustrated by the following equation:

\[
Y = \beta_0 + \sum_{i=1}^{m} \beta_i X_i + \sum_{i=1}^{m} \beta_i^2 X_i^2 + \sum_{i=1}^{m-1} \sum_{j=2}^{m} \beta_{ij} X_i X_j, (i \neq j). \quad (3)
\]
where \( Y \) is the predicted response value, \( \beta_0 \) is the intercept value, \( X_1 \) and \( X_j \) are the independent variables, \( \beta_1 \) and \( \beta_j \) are the linear, interaction and quadratic coefficients, separately. The analysis of variance was adopted to investigate the effects of different variables and to assess the suitability of the model. In addition, the response surfaces were conducted for the experimental data to reveal the roles of single factors and the interactions between these variables. The leaching conditions of copper were also optimized and estimated according to this optimization model.

2.4. Scanning electron microscopy

The residue under optimum leaching conditions were filtered, washed and dried in oven at 40\(^\circ\). Both the residue and raw ore were platinum coated in a sputter coater for preparing samples, more details can be found in the reference [25]. A scanning electron microscope (Philips XL30ESEM-TMP) under an accelerating voltage of 20 kV was conducted to investigate the micro morphology of these samples.

2.5. Structure analysis

The samples before and after extraction were prepared under the optimum leaching parameters. The 1.0000 g of copper ores were initially heated under the vacuum oven to remove the air and moisture. Then the nitrogen was given to the test tube under the required pressure and removed subsequently to evaluate the specific surface area and micro pore structure using the specific surface aperture analyzer (JW-BK112).

3. Results and discussion

3.1. Response analysis

The results of copper leaching efficiency using central composite design (CCD) were shown in Table 3. As can be seen from it, the extraction of copper ranged from 46.52% to 95.25% under the various leaching conditions.

To validate the optimization model, the analysis of variance of response surface methodology (RSM) was conducted and shown in Table 4. The F-Value is widely used to estimate the effects of variables, the higher of the F-Value meaning the greater effect of the variable on leaching efficiency. As shown in Table 4, the initial acidity was the most significant parameter for the copper leaching efficiency with the highest F-value of 133.31 while the leaching time and liquid/solid ratio presented much less significant with a F-value of 27.44 and 13.36 respectively. Note that there was only a 0.01 % chance that an incorrect “F-Value” could be occurred by the noise. For a selected model, adequate precision measured the ratio of signal to noise, which implied the accuracy of the model if this value was larger than 4. The ratio of 18.54 in this study indicated an adequate signal, meaning this model can be utilized effectively to navigate the design space [26].

The leaching parameters can also be estimated by the P-value which is the probability of “Prob>F” in Table 4. When the values of “Prob>F” are less than 0.05, it means that the model terms are significant, and the quadratic regression equation is of high accuracy. Thus, \( X_1 \), \( X_2 \), \( X_3 \), \( X_1 X_2 \), \( X_1 X_3 \), \( X_2 X_3 \), \( X_1^2 \) and \( X_2^2 \) are significant factors for the copper leaching efficiency whereas the others are insignificant. Based on the response surface fitting, the quadratic regression equation for copper leaching efficiency yielded

\[
Y = 88.45+10.87X_1+4.93X_2+3.44X_3-5.11X_1X_2-2.92X_1X_3
-3.38X_2^2-6.15X_1^2. \tag{4}
\]

where \( Y \) is the response value of copper leaching efficiency. \( X_1 \) and \( X_2 \) represent the initial acidity, leaching time and liquid/solid ratio respectively. The coefficient of determination \( R^2 \) for Eq. (4) was 0.96, demonstrating the predicted copper leaching efficiency is in the highly accordance with the experiment results.

The comparison of predicted and experimental copper leaching efficiency was shown in Fig. 1. As can be seen from it, all the points of experimental results were inconsistent with
Table 3 – Experimental design and obtained results.

<table>
<thead>
<tr>
<th>Run</th>
<th>Initial acidity (Kg/t)</th>
<th>Leaching variables</th>
<th>Leaching efficiency/(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>55.20</td>
<td>2.00</td>
<td>3.00</td>
</tr>
<tr>
<td>2</td>
<td>36.80</td>
<td>1.32</td>
<td>4.50</td>
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<td>3</td>
<td>18.40</td>
<td>2.00</td>
<td>6.00</td>
</tr>
<tr>
<td>4</td>
<td>36.80</td>
<td>3.00</td>
<td>4.50</td>
</tr>
<tr>
<td>5</td>
<td>36.80</td>
<td>3.00</td>
<td>4.50</td>
</tr>
<tr>
<td>6</td>
<td>67.74</td>
<td>3.00</td>
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<td>3.00</td>
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<td>9</td>
<td>36.80</td>
<td>3.00</td>
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<td>1.98</td>
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<td>3.00</td>
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<td>17</td>
<td>36.80</td>
<td>4.68</td>
<td>4.50</td>
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<td>4.50</td>
</tr>
<tr>
<td>19</td>
<td>18.40</td>
<td>2.00</td>
<td>3.00</td>
</tr>
<tr>
<td>20</td>
<td>55.20</td>
<td>4.00</td>
<td>3.00</td>
</tr>
</tbody>
</table>

Table 4 – Analysis of variance for the response surface methodology.

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of squares</th>
<th>Degree of freedom</th>
<th>Mean square</th>
<th>F-Value</th>
<th>Prob &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>X₁</td>
<td>1612.19</td>
<td>1</td>
<td>1612.19</td>
<td>133.31</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>X₂</td>
<td>331.84</td>
<td>1</td>
<td>331.84</td>
<td>27.44</td>
<td>0.0004</td>
</tr>
<tr>
<td>X₃</td>
<td>161.63</td>
<td>1</td>
<td>161.63</td>
<td>13.36</td>
<td>0.0044</td>
</tr>
<tr>
<td>X₁X₂</td>
<td>208.59</td>
<td>1</td>
<td>208.59</td>
<td>17.25</td>
<td>0.0020</td>
</tr>
<tr>
<td>X₁X₃</td>
<td>68.39</td>
<td>1</td>
<td>68.39</td>
<td>5.65</td>
<td>0.0387</td>
</tr>
<tr>
<td>X₂X₃</td>
<td>91.60</td>
<td>1</td>
<td>91.60</td>
<td>7.57</td>
<td>0.0204</td>
</tr>
<tr>
<td>X₁²</td>
<td>545.60</td>
<td>1</td>
<td>545.60</td>
<td>45.12</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>X₂²</td>
<td>17.18</td>
<td>1</td>
<td>17.18</td>
<td>1.42</td>
<td>0.2608</td>
</tr>
<tr>
<td>X₃²</td>
<td>0.79</td>
<td>1</td>
<td>0.79</td>
<td>0.07</td>
<td>0.8036</td>
</tr>
<tr>
<td>Residual</td>
<td>120.93</td>
<td>10</td>
<td>12.09</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

R² = 0.96; Rₘₐₜ² = 0.93; adequate precision = 18.54(>4).

Fig. 1 – Predicted vs actual leaching efficiency.

The linear prediction, proving the accuracy of the quadratic model. The coefficient of determination is 0.962.

3.2. Optimization of leaching process

Three-dimensional surface plots were drawn between two experimental factors while holding another variable at its central level to demonstrate the influence of variables on the copper leaching efficiency. As shown in Figs. 2 and 3, the 3D curves in the x–y plane presented the effects of independent variables on the dependent factor.

Fig. 2 showed the three-dimensional curves of temperature and initial acid content on the copper leaching efficiency. As can be seen from it, the copper leaching efficiency rose dramatically with the increasing initial acidity. When the initial acidity increased from 18.40 to 55.20 Kg/t, the copper leaching efficiency soared markedly from 46.52% to 91.42%. The stirring and chemical reaction between lixiviant and ore facilitated the formation of cracks and pores on the surface and inside the
Fig. 2 – Effects of the initial acidity and leaching time on the copper leaching efficiency (liquid/solid ratio: 4.5).

Fig. 3 – Effects of the initial acidity and liquid/solid ratio on the leaching efficiency (leaching time 3 h).

sample, through which the sulfuric acid diffused and formed the copper sulfate.

With the increase of initial acidity in the solution, more \( \text{H}^+ \) could be participated inside the copper ores via the chemical reaction. The lixiviant diffused through the cracks and pores of the copper ores, reacting with the copper oxides to form the copper sulfate which diffused back to the bulk solution afterwards. The increase of initial acidity can not only increase the concentration of \( \text{H}^+ \), rendering more \( \text{H}^+ \) participate in the leaching process, but also alleviate the mass transfer resistance, facilitating the sufficient contact between leaching agent and copper oxides.

The effects of leaching time on the leaching efficiency was relatively weak [27]. A remarkable rise of the leaching efficiency was noticed along with the extension of leaching time at low initial acidity, whereas at higher initial acidity it was insignificant. At higher initial acidity, both the mass transfer diffusion rate and the chemical reaction velocity were higher, which may cause the less influences of leaching time on the leaching efficiency [28].

Fig. 3 demonstrated the effects of the initial acidity and liquid/solid ratio on the leaching efficiency. It is obvious that the increase of initial acidity as well as liquid/solid ratio give rise to the increase of leaching efficiency. A liquid/solid ratio of 3 was selected in the beginning of this study, led to a viscous slurry with the little mass transfer between the solution and sample. Therefore, a relatively high liquid/solid ratio of 4.5 was chosen to conduct the experiment consequently. When the liquid/solid ratio was low, a lump was easily formed in the pulp with high viscosity, led to the low mass transfer rate and the insufficient interaction between the leaching agent and sample, thereby decreased the low copper leaching efficiency. With the increase of liquid/solid ratio, the viscosity of the pulp decreased markedly which give rise to the high mass transfer rate and the sufficient interaction between the leaching agent and sample, resulting into the increase of leaching efficiency. It is interesting that the increase of liquid/solid ratio from 3 to 6 did not bring a prominent increase of leaching efficiency, as the influences of leaching time and initial acidity. A relatively low liquid/solid ratio commonly selected in the practice to reduce the cost of subsequent electrowinning.

To maximize the leaching efficiency, the optimum leaching parameters were identified by the Design-Expert statistical software. As reported in Table 5, The optimal leaching parameters were the initial acidity of 53.22 Kgt/l, leaching time of 2.54 h and liquid/solid ratio of 5.95, leading to an experimental leaching efficiency of 96.74% which was consistence with the predicted value of 96.59%.

3.3. Characterization of copper ores before and after leaching process

The composition of copper ores before and after leaching were detected by the XRD and shown in Fig. 4. As can be seen from it, the diffraction peak of cupric oxide (CuO), cuprite (Cu₂O) and malachite (Cu₂(OH)₂CO₃) in the samples after leaching were distinctly weakened, indicating a large proportion of copper components was removed through reacting with the raffinate. The remaining copper components in the leaching residue should be the chalcocite (Cu₂S) that was difficult to be dissolved in the sulfuric acid solution. The key reactions occurred during the leaching process can be summarized as:

\[
\text{CuO} + \text{H}_2\text{SO}_4 = \text{CuSO}_4 + \text{H}_2\text{O} \quad (5)
\]

\[
\text{Cu}_2\text{O} + \text{H}_2\text{SO}_4 = \text{CuSO}_4 + \text{Cu} + \text{H}_2\text{O} \quad (6)
\]

\[
\text{Cu}_2\text{(OH)}_2\text{CO}_3 + 2\text{H}_2\text{SO}_4 = 2\text{CuSO}_4 + \text{CO}_2 + 2\text{H}_2\text{O} \quad (7)
\]

The size ranges of copper oxides before and after being leaching were characterized by sieving test and shown in Fig. 5. As is shown in Fig. 5, the content of coarse particles decreased obviously while fine particles increased markedly after the leaching process. Specifically, the particles in the samples with size range of >105 um were decreased from 18.96% to 0.58% after leaching. Likewise, the contents of particles with the size ranges of 75–105 μm and 38–75 μm were reduced after leaching. However, for the percentage of fine particle with size range of <38 um, the content of which after leaching was largely increased from 43.71%–85.00 %. The removal of copper components from copper ores through reacting with the sulfuric acid solution resulted into the finer particle size of the copper ores.

To investigate the surface morphology of leaching residue, the SEM analysis of samples before and after leaching was demonstrated in Fig. 6. As shown in Figs. 6(a) and (b), the sur-
Table 5 – Optimum leaching parameters and comparative leaching efficiency.

<table>
<thead>
<tr>
<th>Initial acidity, Kg/t</th>
<th>Leaching time, h</th>
<th>Liquid/solid ratio</th>
<th>Leaching efficiency,%</th>
</tr>
</thead>
<tbody>
<tr>
<td>53.22</td>
<td>2.54</td>
<td>5.95</td>
<td>96.74</td>
</tr>
</tbody>
</table>

Fig. 4 – XRD diagram of samples before and after leaching.

The face of mineral particle before leaching is flat. After leaching, many pores and cracks could be obviously observed in mineral particles as shown in Figs. 6(c) and (d). The erosion feature after leaching process could also be found in Fig. 6(d). The pores and cracks on the ore surface made the copper minerals exposed outside and facilitated the diffusion of leaching agent, thereby promoting the leaching efficiency.

Fig. 5 – Particle size ranges of copper oxides before and after leaching.

To further identify the cracks and pores of the copper ores after leaching, the nitrogen adsorption-desorption isotherms at 77 K of the sample before and after leaching process were conducted and shown in Fig. 7 [29,30]. As can be seen from it, the nitrogen adsorption volumes of copper ores after leaching were obviously higher than that before leaching. The specific

Fig. 6 – SEM images of copper ores before and after leaching: (a) and (b) before leaching; (c) and (d) after leaching (leaching conditions: initial acidity of 53.22 Kg/t, leaching time of 2.54h and liquid/solid ratio of 5.95).
surface area and adsorption mean aperture of copper ores before leaching were 1.34 m²·g⁻¹ and 8.49 nm, whereas those of samples after leaching were increased to 2.28 m²·g⁻¹ and 9.42 nm, respectively. It revealed the surface area of copper ores was largely improved after leaching, indicating the existence of well-developed micro-cracks and micro-pores of the copper ores which could be attributed to the extraction of encapsulated copper.

4. Discussions

In this study, the raffinate from the extraction process was successfully recycled and utilized as the leaching agent for the copper leaching, the operating conditions were optimized by the RSM. A copper leaching efficiency of 96.74 % was achieved under the optimum leaching parameters, which could largely reduce the consumption of acid. The analysis of copper ores before and after leaching found that the micro pores and cracks occurred in the particles after leaching, the specific surface area and adsorption mean aperture of samples after leaching increased as well, which could be attributed to the removal of copper. An illustration diagram of the selective leaching process of copper ores using raffinate as the leaching agent was shown in Fig. 8. Originally, all the minerals were coexisted in the copper ores in which the copper oxides were act as the “glue”. After the addition of raffinate, the copper oxides were dissolved and the smaller particles with cracks and pores were produced.

Currently, there are many ways to recover metal ions from aqueous solution, such as electrocoagulation [31-33] and adsorption [34]. However, the electrocoagulation has the limitation due to its high energy cost, and the adsorption needs extra adsorbing materials which may cause secondary pollution. The present study proposed a reasonable way to deal with the raffinate by using it as the leaching agent in the copper leaching process. The metal ions of raffinate can be flow into the extraction process after leaching so that the copper ions of raffinate can be recovery. In this way, both the high concentration of acid and copper ions can be fully used. Note that the other metal ions of raffinate may have disadvantage of extraction process, thereby the extract agent as well as the operating conditions of extraction process need to be carefully selected.

5. Conclusions

The raffinate from the extraction process were firstly cyclic utilized for the copper leaching in this study. The operating parameters including initial acidity, leaching time, and the liquid/solid ratio were investigated by the module of central composite designs in the response surface methodology (RSM) to achieve the optimal leaching conditions. The initial acidity was found to have the significant effects on the copper leaching efficiency while the other variables presented minor effects. The optimum leaching parameters were confirmed as the initial acidity of 53.22 Kg/t, leaching time of 2.54 h and liquid/solid ratio of 5.95, which resulted into a maximum leaching efficiency of 96.74 %. Further explorations revealed that the micro-cracks and micro-pores were well-developed in the copper ores during the leaching process, which led to the decrease of particle size and the improvement of leaching performance.

Fig. 7 – Nitrogen adsorption-desorption isotherms at 77 K of copper ores before and after leaching.

Fig. 8 – The illustration diagram of the selective leaching process using raffinate as the leaching agent.
Conflict of interest statement

We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service and/or company that could be construed as influencing the position presented in, or the review of, the manuscript entitled“The optimization and characterization of the recycling utilization of raffinate in the copper leaching process”.

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